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Influence of process parameters on Electrodeposited Ni-Al_2O_3 composites by conventional and sediment type of co-deposition by Grey Scale Analysis

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Abstract— **Ni-Al_2O_3 composites are prepared over a mild steel specimens using electrodeposition method. The coatings are prepared by conventional and sediment type depositions using watts type nickel with Al_2O_3. Three primary plating parameters are considered for electro-deposition, namely current density, temperature of the bath, particle concentration in bath with three levels for both conventional and sediment type depositions. L-9 orthogonal array of taguchi design is chosen for experimental design. The deposited coatings are examined with optical microscope, SEM micro graphs, XRD and EDX investigations to confirm the deposition. The influence of parameters on micro hardness of electro-deposition was investigated with signal to noise ratio analysis for both conventional and sediment type deposition. It is inferred that current density and temperature of the bath has a sound effect on micro hardness of coating by conventional type of deposition whereas temperature of the bath and particle concentration has a major effect on micro hardness of coating by sediment type of deposition. Grey scale analysis was employed to indentify the significance of process parameters and optimization of process parameters for the better micro hardness values.**

Index Terms— **Composite coating, Electro deposition, micro hardness, S/N ratio.**

I. INTRODUCTION

Composite coatings are prepared by various techniques like physical vapour deposition, chemical vapour deposition, electrocodeposition and thermal spraying. Electro deposition has been identified as the best method based on the advantages such as ease of control of process parameters, rate of uniform deposition, low cost of experimental setup, good reproducibility and can be prepared in room temperature and normal pressure conditions.

There are various models that have reported the formation of electro co-deposited composite coatings. The first model accounts for electro deposition of metals with particles, which was proposed by Guglielmi. According to investigations, electro codeposition proceeds in two steps. In the first step, the particles were weakly attached on the surface of the cathode by Van der Waals weak forces with high degree of surface coverage. Then, the weakly held particles are trapped in the deposit due to adsorption behaviour by Coulomb force which leads to deposition of particles on the cathode surface [1].

Yet another establishment was described by Celis [2] for the process of electro co-deposition and proposed basic five-steps; i) the particles were surrounded by ionic cloud like formation ; ii) using convection mode of reinforcement, their occurs a mass transfer of the particles along the hydrodynamic boundary layer ; iii) on account of diffuse, their occurs a mass transfer of particles along the cathodic area of deposition, iv) on the cathode, due to the magnetism effect of the electro active and free ions gets adsorbed on the particles, v) finally, diminishing the electro effect of these adsorbed ions on the cathode and also incorporation of the absorbed ions to develop into a metal matrix. Yahia and Adel [3] demonstrated the reasons for the absorption of these particles in the matrix metals and stated that: i) huge amount of the particles were transferred from the electrolytic solution to the cathodic surface ii) due to the electrically charged cathodic action, adsorption of the particles takes place, iii) these adsorbed particles then disperse into the formation of a growing metal matrix layer. The basics of the formation of the electro composites were also supported by Vereecken [4] who gave a model for the electro co-deposition process subsided by the deposition of the particles in the matrix. Another developer named Fransaer who proposed yet another model for the same.

Aruna [5] had examined and have also proposed on the naturally penetrated alumina particles in Nickel matrix and tells that to improve the deposited metal matrix composites, utilisation of porous particles could anyway lead to the betterment as it would improve the nature of adhesion and micro hardness of the particles and these properties solely depends on the bulk and the dissemination of the particles within the metal matrix. Acceptable quantity of these particles would increase the micro hardness, wear resistance, corrosion resistance properties of the coatings.

Haifeng Liu [6] had prepared a Al-Ni composite which constituted an amount of 35% of Al content in the dispersed composite and these dispersion of the particles accounted for the fraction of the amount of Al dispersed during the deposition.

The electro co-deposition process are primarily affected by properties such as the density of current, plating bath concentration of ceramic particles, pH scale and the temperature of the bath and was suggested by Narasimman [7]. They also investigated that considerable improvement in the mechanical properties such as micro hardness of coating, surface texture, scratch resistance and wear resistance can be achieved by dispersion of second phase materials in metal matrix by electrocodepostion.

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Ni-Fly ash composite coatings [8] were prepared by electrodeposition method by adjusting process parameters in order to identify the micro hardness of the coating, revealed that the coatings produced higher micro hardness values than pure nickel coatings (198 HV). Saha and Khan have prepared electrodeposited Ni- Al_2O_3 composite coatings by adjusting the process parameters. They investigated the effect of applied current on alumina deposition in nickel matrix [9]. Suman and sahoo [10] developed an electro less Ni-B composite coating by means of electrodeposition. They employed orthogonal array for their experimental design. They considered four process variables such as temperature of bath, reducing agent concentration, nickel concentration and annealing temperature for the experimental study. The micro hardness of coating were investigated and the effects of process parameters on micro hardness of coatings were analysed with the assistance of S/N ratio analysis and ANOVA, identified that annealing temperature and concentration of reducing agent are the main influencing parameters on micro hardness.

In this investigation, an attempt has been made for the preparation of Ni-composite coating with Al_2O_3 particles suspended in watts bath. By adjusting the electrodeposition process parameters the experiments are conducted. L-9 orthogonal array of taguchi approach is implemented for experimental design. The experiments are conducted by both conventional and sediment type electrodeposition process.

II. APPARATUS AND METHODS

The schematic diagram of electroplating setup is shown in figure 1.The equipments used for electro-deposition along with the specifications is given in Table 1. Electro deposition were carried out in a 2000 ml Borosil glass container and a Watts nickel plating bath (Table 4) was employed. The specimen take into consideration for electro deposition was Mild steel plate which has a specification of 74×25.4×1 mm³ was taken as a cathode substrate and area that was considered for deposition was 25.4×25.4 mm² and the remaining area were shrouded. A pure nickel plate was taken as an anode. The mild steel plate was first degreased by acetone and then, polished with dry cloth buffing wheel, for removal of rust from the plating area. Al_2O_3 composite powder (10µm average) was assorted in nickel solution. The plating solution with Al_2O_3 composite powder was agitated for 3 hours for homogeneous blend. All the mild steel cathode plates were etched in anodic and cathodic cleaning bath for removing the surface contamination in the plating area so as to ensure better adhesion of coating. The Mild steel plates are then rinsed with distilled water and kept immersed in plating bath. Figure 1 shows the schematic of electrodeposition setup. The figure 3 represents the conventional and sediment type electrodeposition methods.

Table 1 Equipment used for electro-deposition

Equipment name	Specification
1. Regulated D.C. powder supply	Capacity: 0-30V and 0-2A
2. Hot plate with temperature controller	AC type, 230 volt, 50 Hz, temperature range: 30° to 110°C
3. Thermocouple	‘K’-type
4. Mechanical stirrer speed regulator	Range: 0-1200 rpm
5. pH meter	Range: 1-14

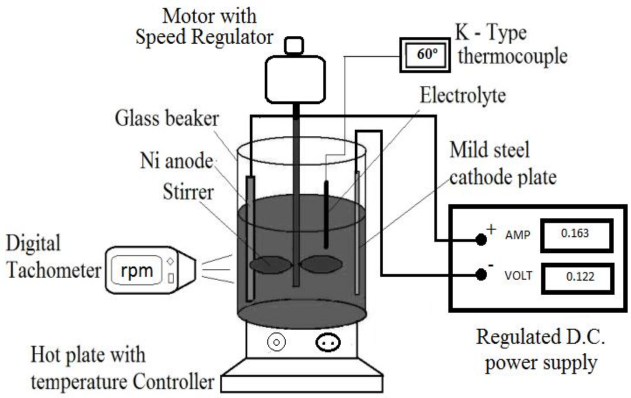

Figure 1 Schematic Diagram

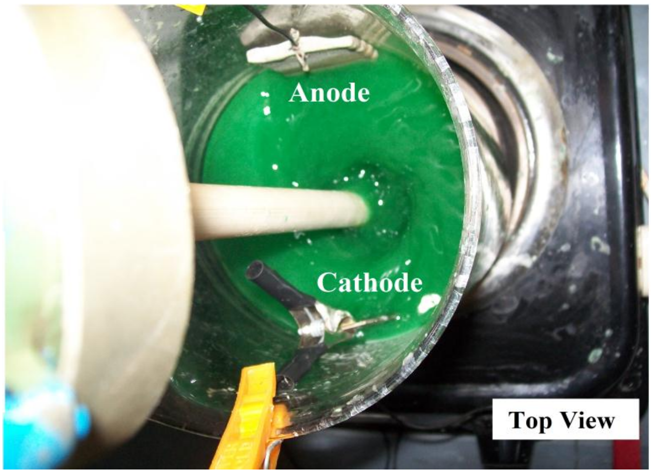

Figure 2 Photograph - During composite plating

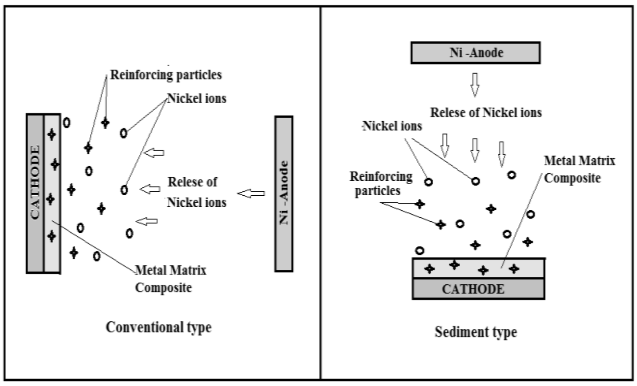

Figure 3 Conventional type and sediment type electro-deposition

Table 2

Experimental factors	Symbols
Current density – A/dm^2	Factor A

Bath temperature - °C	Factor B
Particle concentration in bath –g/l	Factor C

Table 3

Fixed parameters	
Electrolyte level	1000ml
Time for plating for all conditions	60 minutes
Position of anode and cathode	For conventional type - Vertical For sediment type - Horizontal
Type of current applied	D.C.- Direct

Table 4 Constituents of Watt's bath

Constituents	Concentration, g/l
Nickel sulphate	250
Nickel chloride	30
Boric acid	40

The particles that were to be coated were kept in suspension by a motorized stirrer and the speed was observed by digital tachometer and was controlled by a speed controller unit. A regulated D.C power supply machine (manufactured by Royal Instruments, India, capacity: 0-30V and 0-2A) was used for power supply to the circuit. The pH of the electrolyte was adjusted using pH meter and was controlled by the use of diluted acidic or basic solutions. A hot plate with temperature controller unit was used for heating up the bath. A 'K'-type thermocouple was used to observe the temperature of the bath. Ni anode and mild steel cathode was separated by a constant distance and the time for which the process was carried was taken to be 60 minutes for all cases. pH was taken as 4 for all cases. Electrodes were placed vertically for conventional type and horizontally for sediment type (figure 3). While plating Ni ions from anode captured the inert particles in the solution and co deposited the same on the cathode substrate and hence the composite coating. The experiment was done by L9 orthogonal array and nine samples were separately prepared for each type of deposition. Table 2 shows the parameters considered for carrying out the investigation. Table 3 shows the parameters that were kept fixed throughout the process.

III. RESULTS AND DISCUSSIONS

A. Assessment of surface morphology, volume fraction of Al_2O_3 of Ni –Al_2O_3 composite coating

The coated samples were polished and cleaned for investigation. The pattern of deposition was examined by scanning electron microscope. Volume percentages of embedded alumina particles were determined from optical micrographs using high transmission metallurgical microscope. It was found that the particles were dispersed uniformly in nickel matrix that was prepared from conventional type of deposition and also from sediment type of deposition. This confirms the deposition of nickel and ceramic particles in the metal matrix.SEM micro graphs of Ni- Al_2O_3 composites is shown in figure 6.

The XRD (Figure 4) and EDAX (Figure 5) were used to analyse the compositional phases in the coated samples. This also confirms the presence of nickel and Al_2O_3 phases in the deposited layers.

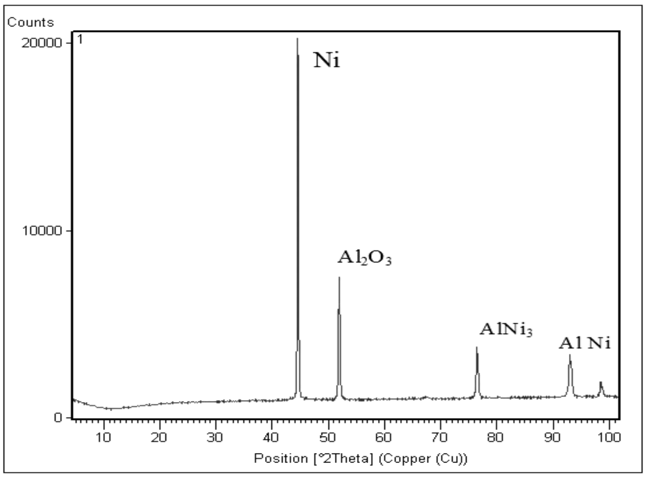

Figure 4 – XRD pattern of Ni-Al_2O_3 composite

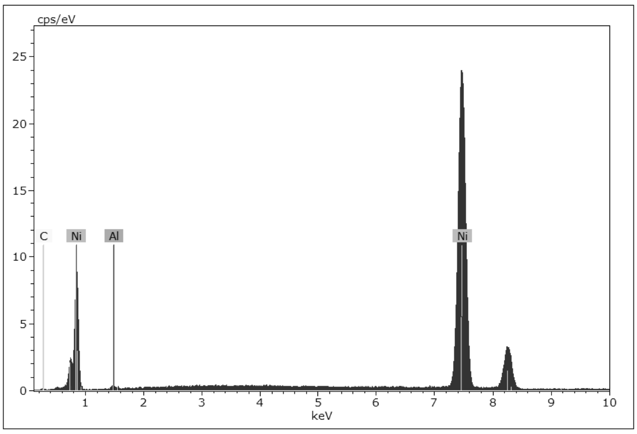

Figure 5 – EDAX data for Ni-Al_2O_3 composite

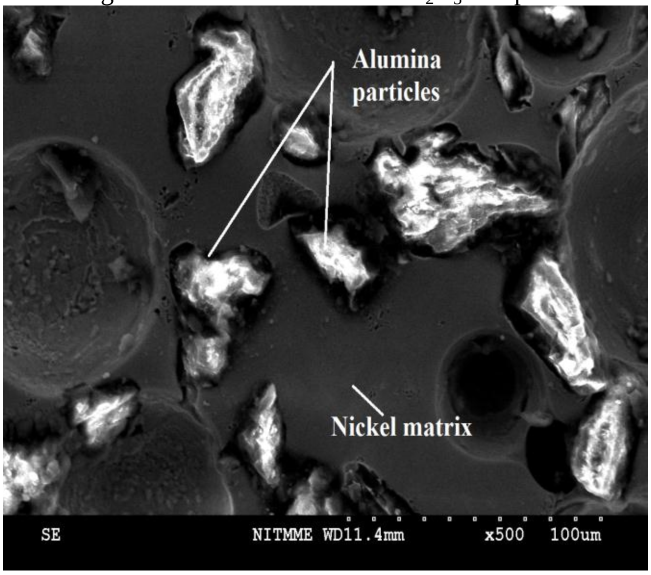

Figure 6 SEM micrographs of Ni- Al_2O_3 composite coating

B. Assessment of micro hardness of Ni –Al203 composite coating

Micro hardness of the coated samples were examined in Vickers micro hardness tester (SHIMADZU-TYPE HMV-1/-2) with a payload of 100 gram of force for 10s of indentation period. It was focussed with 400X magnification. The micro hardness was calculated by the digital readout. Micro hardness for each sample was examined with three trials and the average value was considered.

C. Analysis of S/N ratio

For determining the effect of each variable on the output, the signal-to-noise ratio or the SN number, has to be calculated for each experiment [11, 12, 13]. In this design, micro hardness is the responsible variable which needs to be maximum and hence 'larger the better' approach is used.

S/N ratio (larger the better) for the maximization of micro hardness is calculated using the formula,

$$\frac{S}{N} = -10\log\left[\frac{\sum_{i=1}^{n}\left[\frac{1}{{y_i}^2}\right]}{n}\right]$$

Table 5 process parameters and levels

Parameters	Unit	Levels		
		1	2	3
A.Current density	A/dm²	1	2	4
B.Temperature of bath	˚C	30	45	60
C.Bath concentration	g/l	15	30	45

Table 6 Experimental responses of conventional type deposition

S.no	Current	Temp	Bath con.	Hardness (HV)	S/N ratio
1	1	1	1	280	48.9432
2	1	2	2	216	46.6891
3	1	3	3	265	48.4649
4	2	1	2	428	52.6289
5	2	2	3	1019	60.1635
6	2	3	1	1292	62.2253
7	3	1	3	481	53.6429
8	3	2	1	1671	64.4595
9	3	3	2	701	56.9144

Table 7 Experimental responses of sediment type deposition

S.no	Current	Temp	Bath con.	Hardness (HV)	S/N ratio
1	1	1	1	358	51.0777
2	1	2	2	2389	67.5643
3	1	3	3	242	47.6763
4	2	1	2	943	59.4902
5	2	2	3	295	49.3964
6	2	3	1	381	51.6185
7	3	1	3	662	56.4172
8	3	2	1	2543	68.1069
9	3	3	2	583	55.3134

Table 8 Mean S/N ratio values of responses by conventional deposition

Levels	Level1	Level2	Level3	Delta	Rank
Current	48.02	58.33	58.33	10.31	1
Temp	51.73	57.09	55.86	5.36	3
Bath conc	58.53	52.07	54.08	6.46	2

Table 9 Mean S/N ratio values of responses by sediment deposition

Levels	Level1	Level2	Level3	Delta	Rank
Current	55.43	53.49	59.94	6.45	3
Temp	55.65	61.68	51.53	10.15	1
Bath conc	56.92	60.78	51.15	9.63	2

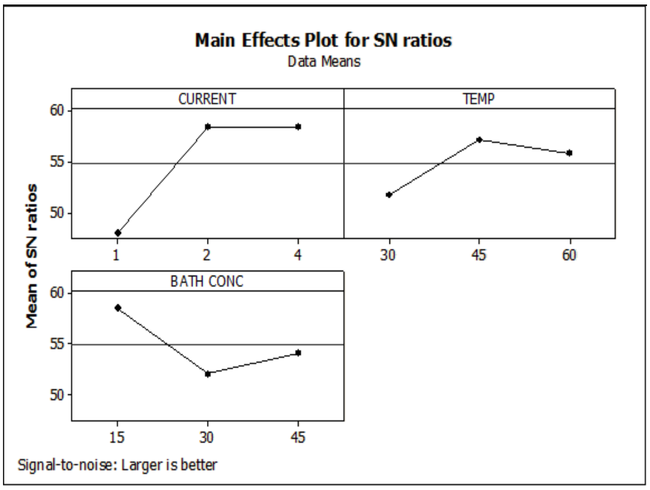

Figure 7 S/N ratio effect Plot For conventional Deposition

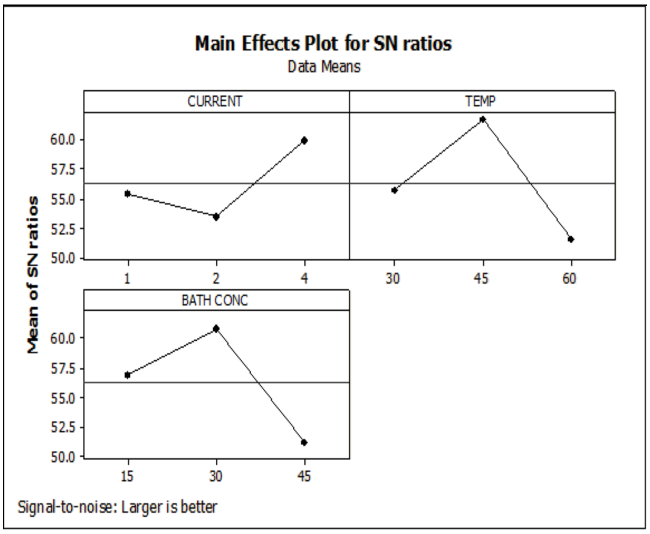

Figure 8 S/N ratio effect Plot For Sediment Deposition

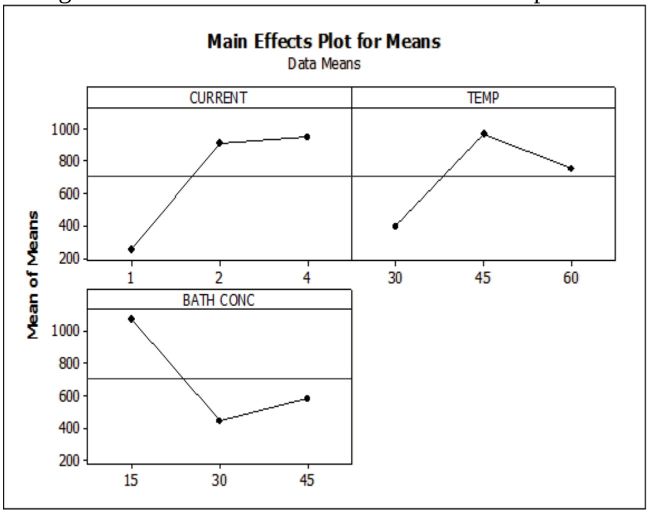

Figure 9 mean effect Plot For conventional Deposition

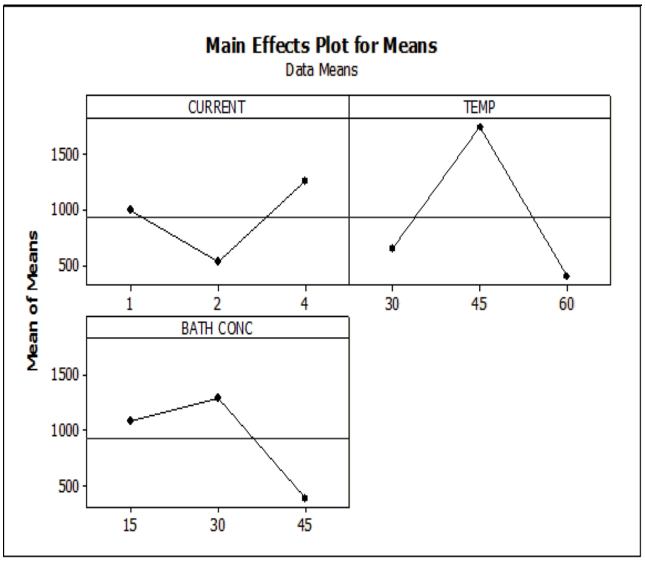

Figure 9 mean effect Plot for Sediment Deposition

MINITAB 16 (software) was employed for determining S/N ratio by design of experiments module. The effect plots were generated for S/N ratio and mean effects. Table 6 shows the experiment outcomes and S/N ratio values for micro hardness by conventional method and Table 7 shows the outcomes and S/N ratio values for the same quantity by sediment method.

Table 8 and table 9 indicates the mean S/N ratio values for each level of control variables for conventional and sediment type of deposition respectively. The tables have a value Delta which is the difference between maximum and minimum S/N ratio values. They are ranked based on largest delta value. Thus, current density is assigned rank 1 in case of conventional and is the most influencing factor as far as micro hardness is considered. Temperature of the bath is ranked 1 in case of sediment type of deposition and is the most influencing factor on micro hardness for sediment type. In order to verify S/N ratio values, grey scale analysis was implemented to identify the significances of each parameter.

D.GREY SCALE ANALYSIS

Grey scale analysis can be implemented for optimising multiple performance characteristics into single grey relational grade. The steps [14,15] to be considered are:

D.1 STEP 1

The S/N ratios obtained by Taguchi's method are normalised in the range of 0-1. The normalised result was obtained from the following equation

$$x_i(k) = \frac{\eta_i(k) - \min \eta_i(k)}{\max \eta_i(k) - \min \eta_i(k)}$$

Where,

$x_i(k)$ Value after the grey relational generation

$\max \eta_i(k)$ Largest value of for $\eta_i(k)$ the kth response

$\min \eta_i(k)$ Smallest value of for $\eta_i(k)$ the kth response

D.2 STEP 2

The $x_o(k)$=1, k=1, 2…9 are the ideal sequence for hardness. Grey relational coefficients is found from deviation, which is nothing but 1-normalised S/N ratio. The grey relational coefficient is found out by the following equation

$$\zeta_i(k) = \frac{\Delta_{\min} + \psi \Delta_{\max}}{\Delta_{oi}(k) + \psi \Delta_{\max}}$$

where,

$\Delta_{oi} = \left\| x_o(k) - x_i(k) \right\|$ Difference of the absolute value $x_o(k)$ & $x_i(k)$

$\Delta_{\min} = \min\limits_{\forall j \in i} \min\limits_{\forall k} \left\| x_o(k) - x_i(k) \right\|$ Smallest value of Δ_{oi}

Largest value of Δ_{oi}

$\Delta_{\max} = \max\limits_{\forall j \in i} \min\limits_{\forall k} \left\| x_o(k) - x_j k) \right\|$

φ Distinguishing coefficient and its value is between 0 - 1. Generally, φ =0.5 is used;

D.3. STEP 3

Grey relational grade γ_i is found out by,

$$\gamma_i = \frac{1}{n} \sum_{k=1}^{n} \zeta_i(k)$$

Where,

n - Number of performance characteristics

Higher the value of grey relational grade, stronger is the relational degree between the ideal sequence and the given sequence. Ideal sequence is the best process response in experimental layout. Higher relational grade insinuates that the corresponding parameter combination is closer to optimal.

TABLE 10 Grey scale analysis for conventional type of deposition

Current	Temp	Bath con.	Hardness	S/N ratio	Normalised S/N ratio	Deviation	Grey relational coefficient
1	1	1	284	48.9432	0.133	0.867	0.365
1	2	2	219	46.6891	0	1	0.333
1	3	3	264	48.4649	0.100	0.9	0.357
2	1	2	426	52.6289	0.334	0.666	0.429
2	2	3	1018	60.1635	0.758	0.242	0.673
2	3	1	1292	62.2253	0.874	0.126	0.798
3	1	3	482	53.6429	0.391	0.61	0.450
3	2	1	1670	64.4595	1	0	1
3	3	2	700	56.9144	0.575	0.424	0.541

TABLE 11 Grey scale analysis for sediment type of deposition

Current	Temp	Bath con.	Hardness	S/N ratio	Normalised S/N ratio	Deviation	Grey relational coefficient
1	1	1	358	51.0777	0.166	0.834	0.374
1	2	2	2389	67.5643	0.973	0.027	0.948
1	3	3	242	47.6763	0	1	0.333
2	1	2	943	59.4902	0.578	0.422	0.54
2	2	3	295	49.3964	0.082	0.918	0.352
2	3	1	381	51.6185	0.192	0.808	0.382
3	1	3	662	56.4172	0.421	0.579	0.466
3	2	1	2543	68.1069	1	0	1
3	3	2	583	55.3134	0.371	0.633	0.442

Table 12 Mean grey scale coefficient values of parameters on micro hardness by conventional deposition

Levels	Level1	Level2	Level3	Delta	Rank
A.Current	0.351	0.633	0.663	0.313	1
B.Temp	0.414	0.668	0.564	0.224	3
C.Bath conc	0.721	0.433	0.493	0.288	2

Table 13 Mean grey scale coefficient values of parameters on micro hardness by sediment deposition

Levels	Level1	Level2	Level3	Delta	Rank
A.Current	0.551	0.425	0.636	0.211	3
B.Temp	0.460	0.766	0.385	0.381	1
C.Bath conc	0.585	0.644	0.383	0.261	2

IV. RESULTS AND DISCUSSION

Table 10 shows the S/N ratio, Normalised S/N ratio, Grey scale coefficient and Grey scale grade. The value of distinguishing coefficient was taken as 0.5. The higher the grey relational grade, better the performance characteristics. It can be inferred that A3B2C1 (experiment no: 7) has the highest grey relational grade and has the best multi performance characteristics. The procedure to find out average grey relational grade is as follows: first, group the grey relational grades by factor level for each column in the orthogonal array and take average of them.

The average grey relational grade for each level is calculated similarly. The greater the value of grey relational grade, the stronger is the correlation to the reference sequence. The optimal process combination is A3B2C1 for conventional type. The values of optimal parameters were current density of 4 A/dm², temperature of the bath of 30 ˚c and bath concentration of 15 g/L. The deviation (difference between maximum and minimum value) is also shown. The maximum value given in table12 is 0.313 for the factor current density. Thus current is the deciding factor in conventional type of deposition.

The optimal process combination is A3B2C1 for sediment type. The values of optimal parameters were current density of 4 A/dm², temperature of the bath of 30 ˚c and bath concentration of 15 g/L. The deviation (difference between maximum and minimum value) is also shown. The maximum value in table 13 is 0.381 for temperature of the bath. Thus temperature of bath is the deciding factor in sediment type of deposition.

V. CONFIRMATION TEST

After completion of analysis, the result must be verified to check the accuracy of the analysis. The predicted relational grade using optimum parameter can be expressed as [14,15],

$$\hat{\gamma} = \gamma_m + \sum_{i=1}^{q} (\overline{\gamma_i} - \gamma_m)$$

Where,

γ_m Total mean grey relational grade

$\overline{\gamma_i}$ Mean grey relational grade at optimum level

The predicted value using the above formula was 1588.88HV whereas the experimental value was 1671HV which had a deviation of 4.9% in the case of conventional type of deposition. The predicted value was 2444.21HV whereas the experimental value was 2543HV which had a deviation of 3.88% in the case of sediment type of deposition. Thus, the results were in agreement with the experiment.

VI. CONCLUSION

Confining it, we have discussed about how Grey relational analysis with Taguchi's L9 orthogonal array makes use of the various characteristics like temperature of bath, bath concentration and current density. Response table helps us to determine the largest maximum and minimum grade of the Grey relational analysis and these values depicts that the value of the current density has the strongest effect out of all the other parameters in conventional type and temperature of the bath in the case if sediment type. Further tests showed that the required optimum combinations satisfy the electro co-deposition process of Ni-Al_2O_3 composites. The Nickel-Alumina composites were obtained from the watt bath using two types of electro depositions. We also noticed that by the use of conventional and sediment type of electro deposition, micro sized alumina particles were dispersed. For a conventional type of electro deposition, the determining factors are the temperature of the bath and current density whereas for a sediment type, the significant factors are temperature of the bath and its concentration. Improvement of the reliability of its experimental investigations could confirm by the scientific and statistical approach which adds to the significance of certain parameters as end properties of experimental responses.

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